Standard Operating Procedure
Spring Sampling

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Page 1 of 14

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1.0 Scope and Application

Instructions presented in this Standard Operating Procedure (SOP) are for collecting representative water samples from flowing spring water bodies. A spring is defined as a place where the water table crops out at the ground surface and water flows out more or less continuously. This SOP describes in detail the spring sampling proposed in the scope of work for the Illinois River Watershed sampling.

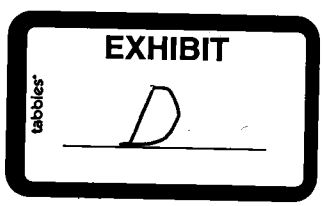
In the Illinois River Watershed, over 50 springs or seeps have been identified. These springs typically result from groundwater seepage or outflow at specific locations during periods of high rainfall. During other times of the year, the groundwater may enter streams as base flow (gaining stream). This task will evaluate the contamination in selected areas by sampling springs. The information will be used to provide a preliminary evaluation of potential groundwater contamination.

Based on the locations of known springs in the United States Geological Survey (USGS) database and locations where various samples have been collected by Universities, approximately 20 springs will be selected for sampling. Samples will be collected and field parameters measured (pH, ORP, SC, DO, and temperature). Samples for dissolved constituents will be filtered in the field using a 0.45 micron filter. The preserved samples will be sent to the laboratories for analyses.

2.0 Sampling Methods Summary

Presented below are sampling instructions for collecting spring water samples. Prior to sample collection, water body characteristics (size, depth, flow) should be recorded in the field logbook. Sampling should proceed from downstream locations to upstream locations so that disturbance from sampling does not affect sampling quality. In collecting spring water samples from any source, care must be taken to minimize disturbance as it is retrieved.

The sampler should not place the sample bottle into the flowing spring water because some sample bottles contain chemical preservatives. Instead, the spring water should be collected in a container (bucket or gallon jug) that has been properly washed and rinsed at each sample location.



2.1 Spring Water Sampling Locations

Based on the locations of known springs in the United States Geological Survey (USGS) database and locations where various samples have been collected by Universities, approximately 20 springs in the Illinois River watershed have been selected for sampling. In order to collect a representative spring water sample, the sample location must be within 200-feet of the spring head (location where the water table outcrops). In the event the spring head is not accessible, the sample location should be hydrologically tied to the location where the water is flowing out of the ground surface.

2.2 Sample Types

The type of sample should be designated when selecting a sampling method. Water samples can either be discrete or composite samples. A discrete sample is defined as a single aliquot from of a specific location at a given point in time. Composites are samples composed of two or more specific aliquots (discrete samples) collected from one or several sampling locations and/or different points in time. This type of sample represents an average value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix.

2.3 Sampler Selection

The choice of samplers is dictated by sampling objectives (surficial versus subsurface samples) and site constraints based on water depth. Each sampling technique presents various advantages and disadvantages for its application. For example, sample disturbance, sample volume, chemical and physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique.

Discrete sediment samples from shallow to moderately deep water can be collected efficiently using polycarbonate (or Lexane) tube. Polycarbonate tube samplers are easy to use, portable, and are a direct method for obtaining sediment samples. The tube is forced into the sediment and then withdrawn using a vacuum/suction technique. Table 1 presents the equipment needed for the spring sampling.

Table 1. List of the minimum equipment required for spring sampling.

Equipment	Amount
Yellow Springs Incorporated (YSI) Meter	1
Peristaltic Pump	1
12 Volt Battery	1
Low Density Polyethylene tubing	Several Feet (disposable)
Pump Head tubing (pure silicon)	Several Feet (disposable)
0.45 micron filters	May use 2 to 3 filters per sampling site
Container (gallon jug or bucket)	2
Field Book	1
GPS with compass to show north	1
60 mL syringes	20
Labels	Enough for each samples bottle
Permanent markers and pens	3 each
Coolers for storing and shipping	Enough to carry and ship samples

2.4 Data to be Collected

At each spring sampling location, a suite of physical variables should be recorded. These variables are intended to locate each sampling point and to quantify factors likely to be associated with collecting a representative sample. Variables may include:

- Water depth.
- Approximate flow rate.
- Distance from spring head to sample location.
- Surrounding land use (i.e. farmland, residential, private vs. public property, etc).
- Sketch of the spring location.
- Photo of the sample location (minimum of two photos per site).
- Presence or absence of algae growth.
- Latitude and Longitude readings.

- Temperature.
- Dissolved Oxygen as a percentage and in milligrams per liter.
- Oxidation-Reduction Potential.
- Conductivity.
- pH.

2.5 Sample Nomenclature

Samples should be labeled according to the given names (i.e. Living Water Springs, July Springs, etc) followed by the date the sample was collected. These names were either issued by the United States Geological Survey (USGS) or by local residents. If a spring is found unexpectedly (located in the field but was not from the USGS database) and the sampler feels it is a good location but has no associated name, the sampler should label the spring by either the name of the resident whose property it is on or other means. As long as there are latitude and longitude readings, the location of the spring shall be known. It is preferred that the date be included along with the spring name in case there are multiple visits.

3.0 Spring Sampling Procedures

There are two ways the sample can be collected. By collecting the sample in a container or if the flow is immediately inaccessible or low flow, the spring water can be pumped through a tube directly into the bottles for non-filtered samples or pumped through a filter for the field filtered samples.

When filtering the spring water sample, it is more convenient to use a battery-operated peristaltic pump. A minimum of 1-gallon of the spring water should be purged before taking any samples. If the flow from the spring is too shallow to submerge a gallon jug or bucket, then the water for both filtered and non-filtered samples must be collected using a pump. Otherwise, the non-filtered samples can be collected directly from the stream.

3.1 Sampling Using the Container Method

1. Before approaching the spring, make an assessment as to whether it is located on private property. If the spring is on private property, the sampler may ask (if it is seemingly a safe area) the landowner for permission onto the property for collecting spring samples.

2. Before sampling at each site, a meter reading (using a YSI meter) must be taken. Once the meter appears to have stabilized (i.e. the values are not changing significantly) document the time and then the parameter (pH, DO%, DO mg/L, temperature, ORP and conductivity). Three meter readings must be taken at each sampling site.

3. Locate the proposed sample location with a GPS unit and take photographs (minimum of two photographs per site).

4. If there is a significant amount of flow, take a bucket or gallon jug and collect the spring water and rinse the bucket several times downstream of the sample location. A gallon jug with the top cut off works well because it is flexible and there is a handle.

5. Grab a homogenous, representative sample of spring water by dunking the bucket or jug into the middle stream of water preferably at the point where the groundwater surfaces or very close to that point. Keep sediment and debris from entering the sample. As stated before, the sample location shall be no more than 200-feet downstream of the point where the water reaches the ground surface.

6. Fill the appropriate bottles by pouring slowly without touching the collection jug to the bottles. Included in these bottles for non-filtered samples will be a one-liter amber glass bottle, Four 250-mL sterile plastic bottles, One plastic bottle with nitric acid preservative, One plastic bottle with sulfuric acid preservative, A one-liter plastic bottle with no preservative, And two 40-mL amber glass with hydrochloric acid preservative. When filling the 40-mL amber glass bottles, allow no room for air bubbles. A complete listing of bottles for spring sampling is presented in this SOP.

7. The container method of collecting the spring water works well for collecting the non-filtered samples; however, for the filtered samples, pumping the spring water (using a peristaltic pump and tubing

connected to a 0.45 micron filter) works best. In the event that the spring is located a distance away from the vehicle (far enough from the road that a hike is required) the spring water may be collected in a container, and then taken back to the vehicle and pumped from the container, through the filter and into the bottles.

8. To pump the spring water from the container and through the filter, extend Low Density Polyethylene (LDPE) tubing into the container.

9. Connect the LDPE tubing to a section of flexible pure silicon pump head tubing and then run the pump head tubing through the pump.

10. Connect the pump to a 12.0 Volt battery using the cable provided in the pump kit. The red clip connects to the positive side of the battery and the black clip connects to the negative side of the battery. The other end of the cable connects to the pump for power.

11. Secure the tubing and then connect the other end into the inlet side of a 0.45 micron filter.

12. Turn the pump on and allow the water to run through the tubing, through the filter and into the sample bottles (only after flushing a minimum of 1-gallon through the pump). The bottles for filtered samples include a one-liter plastic bottle with no preservative, a plastic bottle with nitric acid preservative, and a plastic bottle with sulfuric acid preservative. A complete listing of bottles for spring sampling is presented in this SOP.

13. Label each container by using the spring name as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories and parameters. Include the date, time the sample was collected (military time) and the initials of the sampler.

14. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

15. Complete all chain-of-custody documents and field sheets and record in the field logbook.

16. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water.

17. All other equipment (i.e. tubes for the pump, filters and medical gloves) are disposable. Sample material not sent to the lab for analysis will be disposed in a municipal landfill.

18. Repeat procedure for each sample location.

3.2 Sampling Using the Pumping Method

1. Before approaching the spring, make an assessment as to whether it is located on private property. If the spring is on private property, the sampler may ask (if it is seemingly a safe area) the landowner for permission onto the property for collecting spring samples.

2. Before sampling at each site, a meter reading (using a YSI meter) must be taken. Once the meter appears to have stabilized (i.e. the values are not changing significantly) document the time and then the parameter (pH, DO%, DO mg/L, temperature, ORP and conductivity). Three meter readings must be taken at each sampling site.

3. Locate the proposed sample location with a GPS unit and take photographs (minimum of two photographs per site).

4. For the non-filtered samples, if the spring flow is not readily accessible, it is best to use a pump because the sampler can extend the LDPE tubing into the spring water and use the pump to collect the sample in the appropriate bottles for collecting non-filtered samples (only after flushing a minimum of 1-gallon through the pump).

5. For the field filtered sample, it is more convenient to pump the water directly from the spring, through the filter and then into the appropriate bottles.

6. To pump the spring water, extend the LDPE tubing into the stream flow. Once again, grab a sample approximately midway down not to collect debris or sediment.

7. Connect the LDPE tubing to a section of flexible pure silicon pump head tubing and then run the pump head tubing through the pump.

8. Connect the pump to a 12.0 Volt battery using the cable provided in the pump kit. The red clip connects to the positive side of the battery and the black clip connects to the negative side of the battery. The other end of the cable connects to the pump for power.

9. Secure the tubing and then connect the other end into the inlet side of a 0.45 micron filter.

10. Turn the pump on and allow the water to run through the tubing, through the filter and into the sample bottles (only after flushing a minimum of 1-gallon through the pump). The bottles for filtered samples include a one-liter plastic bottle with no preservative, a plastic bottle with nitric acid preservative, and a plastic bottle with sulfuric acid preservative. A complete listing of bottles for spring sampling is presented in this SOP.

11. Label each container by using the spring name as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories and parameters. Include the date, time the sample was collected (military time) and the initials of the sampler.

12. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

13. Complete all chain-of-custody documents and field sheets and record in the field logbook.

14. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water.

15. All other equipment (i.e. tubes for the pump, filters and medical gloves) are disposable. Sample material not sent to the lab for analysis will be disposed in a municipal landfill.

16. Repeat procedure for each sample location.

3.3 Sampling Using a Syringe

In some cases, the spring location may be quite a distance from the road. In this case, it is not convenient to carry the battery and pump (along with all other supplies) the entire distance.

Also, the container method may not work if the flow is not that great. The following is a procedure to collect spring water samples using a syringe.

1. Before approaching the spring, make an assessment as to whether it is located on private property. If the spring is on private property, the sampler may ask (if it is seemingly a safe area) the landowner for permission onto the property for collecting spring samples.

2. Before sampling at each site, a meter reading must be taken. Once the meter appears to have stabilized (i.e. the values are not changing significantly) document the time and then the parameter (pH, DO%, DO mg/L, temperature, ORP and conductivity). Three meter readings must be taken at each sampling site.

3. Locate the proposed sampling location with a GPS Unit and take photographs (minimum of two per site).

4. Withdraw the spring water using a 60-mL or larger capacity syringe. Push the water back out and do this several times to rinse the inside of the syringe.

5. For the non-filtered samples, relinquish the spring water directly into the appropriate bottle being careful not to touch the tip of the syringe to the bottle.

6. For the field filtered samples, withdraw the spring water, connect the inlet side of a 0.45 micron filter to the tip of the syringe and then gently push the water through and into the appropriate bottles for filtered samples. Do not force the water through too hard because this may puncture a hole in the filter.

7. Label the containers by using the spring name and date system as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories and parameters. Include the date, time the sample was collected (military time) and the initials of the sampler.

8. Store the samples on ice or in the refrigerator until shipment to analytical laboratories.

9. Complete all chain-of-custody documents and field sheets and record in the field logbook.

10. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water.

11. All other equipment (i.e. tubes for the pump, filters, medical gloves and syringe) are disposable. Sample material not sent to the lab for analysis will be disposed in a municipal landfill.

12. Repeat procedure for each sample location.

4.0 Sample Containers, Preservation Techniques, Quality Control

A number of different parameters will be collected for and analyzed at each spring location. Table 2 presents a list of all parameters that may be analyzed and a reference to the standard method for that analysis.

Table 2. Parameters and methods summary for water samples collected at springs within the Illinois River watershed.

Parameter	Method
Total dissolved solids, TDS	160.1
Total suspended Solids, TSS	160.2
Total organic carbon	415.2
pH	150.0
Specific Conductivity	120.1
Total Phosphorous (P), nonfiltered	365.2, persulfate digestion
Total P, ortho/reactive, nonfiltered	365.2, direct
dissolved P total, filtered	365.2, persulfate digestion
Dissolved P, ortho/reactive, filtered	365.2, direct
Nitrogen, nitrate + nitrite	353.3
Total Nitrogen	TKN, 351.3
Nitrogen, ammonium	350.2
TAL Metals, dissolved (filtered)	EPA SW-3050/6010
Dissolved Molybdenum (Mo)	EPA SW-3050/6010
Dissolved Phosphorous (P)	EPA SW-3050/6010
TAL Metals, total (nonfiltered)	EPA SW-3050/6010
Total Molybdenum (Mo)	EPA SW-3050/6010
Total Phosphorous (P)	EPA SW-3050/6010
Sulfate, filtered	375.1

Alkalinity, filtered	310.2
Chloride, filtered	325.1
Bacteria	
Total coliform	SM-9221B
enterococcus	SM-9221F
Fecal coliform	SM-9230B
e-coli	SM-9221F
staphylococcus	MPN
campylobacter	MPN
salmonella	MPN
17α-estradiol,17β-estradiol, estrone, estriol	LC-MS-MS

The following is a list of sample bottles required during collection for all parameters:

- 1- 500 mL plastic with nitric acid, non filtered for total metals and P
- 1- 500 mL plastic with nitric acid, filtered in field for dissolved metals and P
- 1- one L plastic with no preservative, non filtered for TDS, TSS, pH, nitrite+nitrate, ortho-P by 365.2
- 1- one L plastic with no preservative, filtered in field for dissolved ortho-P by 365.2, sulfate, chloride, alkalinity
- 1- 500 mL plastic with sulfuric acid, non filtered for total P by 365.2, TKN and ammonium
- 1- 500 mL plastic with sulfuric acid, filtered for dissolved total P by 365.2
- 2- 40 mL VOA vial with hydrochloric acid, non filtered for TOC
- 1- one L glass, non filtered for estrogen metabolites
- 1- 250 plastic sterile, non filtered for bacteria

4.0 Sample Containers, Preservation Techniques, Quality Control

Either at the end of the week or periodically throughout the sampling, samples will be packed and shipped in coolers to one of several different analytical laboratories (contact information

below) depending on analyses required. Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples will include field blanks, duplicates, and split samples. Duplicates should be collected at least once for every twenty samples.

4.1 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment which is in direct contact with the sample water must be cleaned between each sample collection.

Procedures for decontamination are as follows:

- Rinse all surfaces with de-ionized or distilled water.
- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.

4.2 Laboratory Contact Information

The following is a list of contact information and shipping addresses for all analytical laboratories used for water samples.

Bottles for estrogen metabolites (one liter glass) will be shipped to:

General Engineering Laboratories, LLC
741 Corporate Circle, Suite I
Golden, CO 80401
Contact: Paul Winkler, 720-253-3093
Paul.winkler@gel.com

Bottles for nutrients, metals, P, etc (8 bottles: two 500 ml with nitric, two 500 mL with sulfuric, two 1-L no preservative, two 40 mL VOA with hydrochloric) will be shipped to:

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133

Contact: Scott McKee, 800-264-4522
smckee@allabs.com

Bottles for bacteria analyses (one 250 mL plastic sterile) will be shipped to:

FoodProtech
1414 S. Sangre Road
Stillwater, OK 74074
Contact: Siobhan Reilly, 405-533-7779
reilly@foodprotech.com

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Cole Mackelprang, 858-268-2762
E-mail: cmackelprang@emlab.com
Contact (microbiologist): Mark Wallin, 650-742-8132

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

7.0 References

Oklahoma Water Resources Board (OWRB). 2001. *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Herlihy, A.T. 1998. Water chemistry. pp. 57-65 IN: J.M. Lazorchak, D.J. Klemm, and D.V. Peck (Eds.). Unpublished draft. *Environmental Monitoring and Assessment Program - Surface Waters: Western Pilot Study Field Operations Manual for Wadeable Streams.* EPA/620/R-94/004F. U.S. Environmental Protection Agency, Washington, D.C.